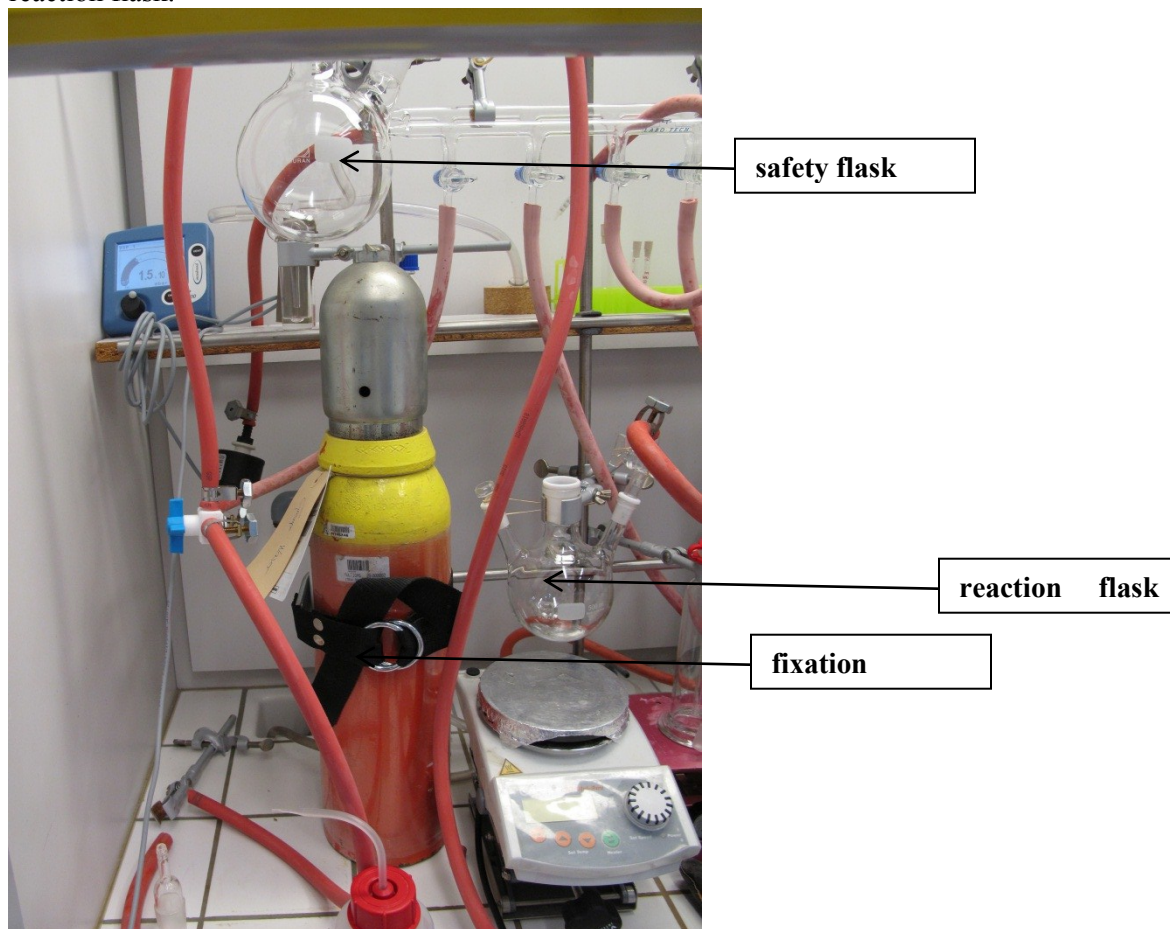


## Working correctly with gas

When working with a reactive gas in a cylinder, there is an inherent risk. Special care has to be taken to allow safe handling. This example is with HCl.

### 1) Fixing the gas bottle.

The first step in all reaction involving gas is a safe fixing of the bottle. In addition, a safety empty flask is added between the gas bottle and the reaction flask. This flask should have a volume larger than the reaction flask.



### 2) Preparation of the glassware

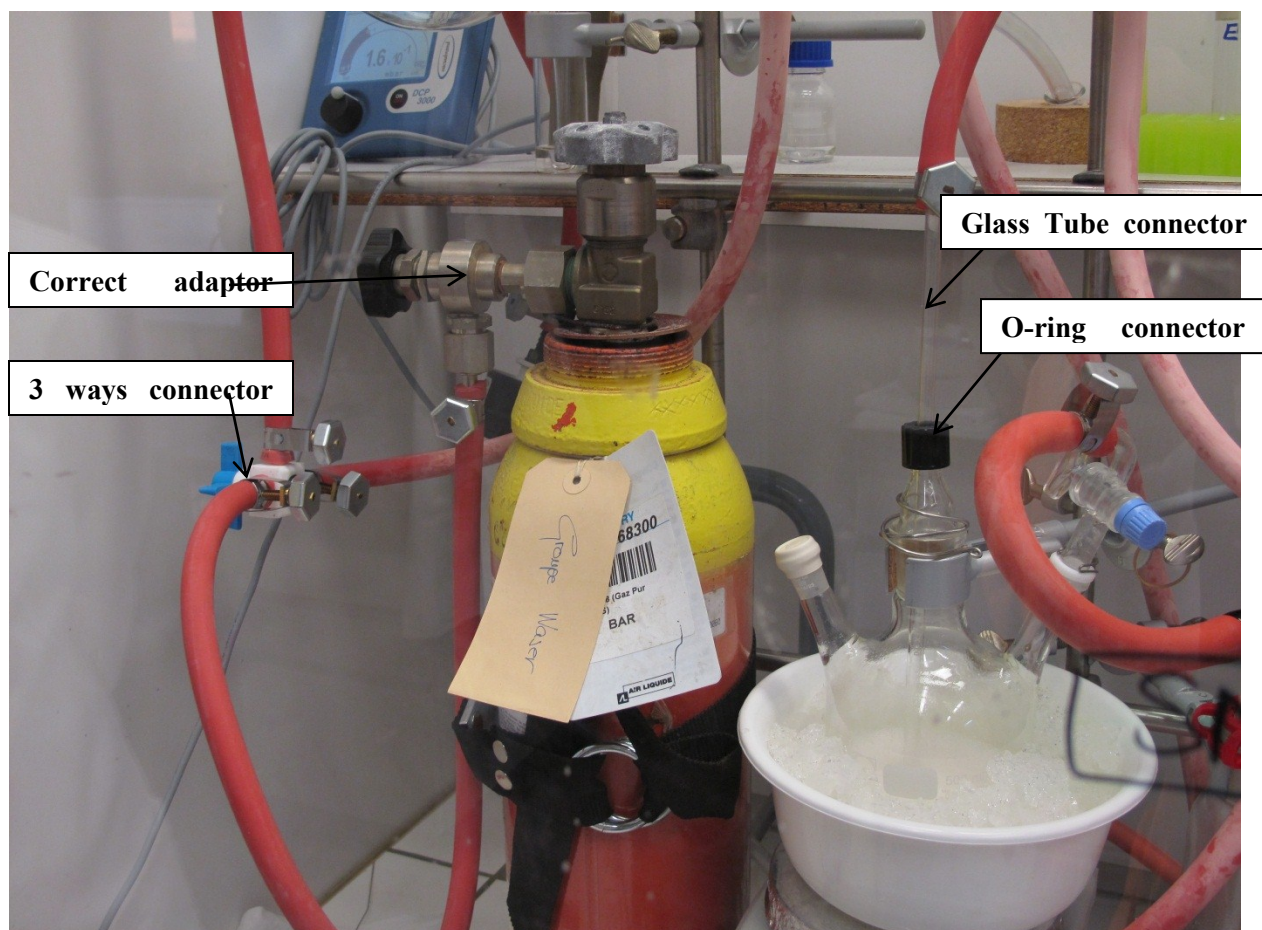
After the safety flask, a three-Neck reaction flask is prepared: two necks for gas inlet and outlet, and one Neck to add solvents and reagents. If the reaction is very water sensitive, it is then connected to two washing bottles containing a drying agent ( $\text{CaCl}_2$  in the case of an acid gas). Finally, two washing bottles are added to neutralize the gas (in the case of HCl, a 4M NaOH solution). **Extreme care has to be taken to connect the washing bottle the right way (see picture):** In no case should the washing liquid be able to be sucked inside the tubing!



## 2) Preparation of the gas connection

In preparation for the gas connection, first put all the glassware under nitrogen. Remove the protecting cap of the bottle and install the correct adaptor (**!!! adaptors are out of different metals depending of the gas, be sure to check with the technician if you have the right one!!!**). Connect the adaptor tightly using the correct tool (hand is not enough). Install a three-way connection just after the bottle: one to the nitrogen line, one to the reaction via the safety flask, one to the gas bottle. In the reaction flask, connect the gas tube to a glass connector equipped with a gas repartition fritte. Use a screwable connector with a O-ring joint to fit the glass tube to the reaction flask. Secure all tubing under pressure with clamps. Test then the complete apparatus with a nitrogen flow.





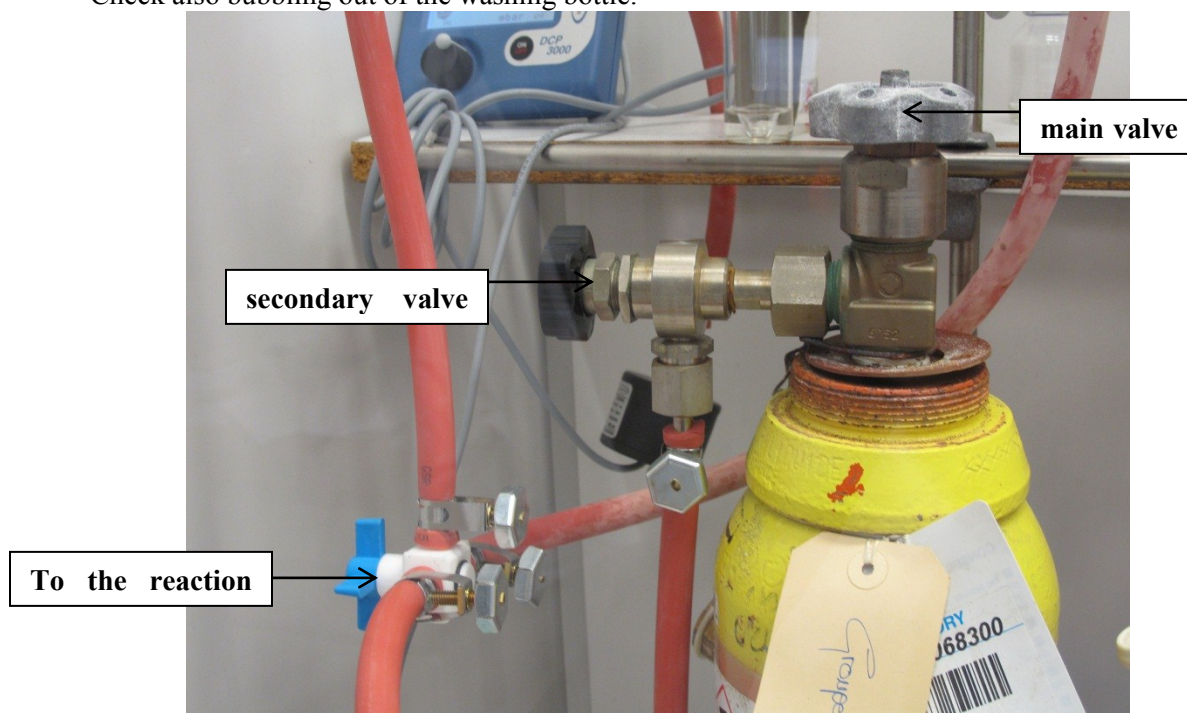
### 3) Adding solvent and reagents

Add solvent or reagents using standard technique. Here a dry solvent was added via canula using the overpressure of an Ar balloon. This can be done true septa via a Teflon tube. The solvent is filtered through a glass fritte during transfer.



### 4) Turn on the gas

- 1) Check that the secondary valve is closed.
- 2) Open the main valve completely. Be ready to close it immediately if the system is not tight.
- 3) Switch the 3-ways connector to connect the gas bottle to the reaction flask (**be careful not to send the gas to the line! Be always careful to have an open system, else there is a big risk of overpressure!**).
- 4) Open slowly the secondary valve on the bottle. Be careful: some valves are not very sensitive, and the gas flow can go from nothing to relatively strong rapidly.
- 5) As soon as the gas come out, insert the gas inlet into the solvent, modulate bubbling with the valve. Check also bubbling out of the washing bottle.



### 5) Monitor the reaction

Check continuously the temperature of the reaction mixture and the washing bottle, if required, cool them down with ice. Check that there is a constant but slow bubbling in the reaction flask and the wash bottle. Check with pH paper if the wash bottle is still basic.





#### 6) Stopping the reaction

- 1) Remove the gas inlet from the solvent
- 2) Close the secondary valve. Close the main valve
- 3) Immediately turn the 3-ways connector to let the nitrogen of the line enter the reaction system.
- 4) Flush with nitrogen for at least 30 min.
- 5) Be careful when removing the adaptor from the bottle (gloves, visage protection) as a tiny amount of gas will still come out. Let the hood on urgency for some time. Screw the protecting cap back on the bottle. Remove the bottle from the hood.